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## V.

CONTRIBUTIONS FROM THE CHEMICAL LABORATORY  
OF HARVARD COLLEGE.

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Presented, July, 1881.

## CRYSTALLINE FORM OF CRYOLITE.

THE crystalline form of Cryolite was described by Dana in his "System of Mineralogy," 1868, as Trimetric, but, as he distinctly stated, the system in which the mineral crystallizes was considered doubtful. Des Cloizeaux has since investigated the optical characters, and determined the system of crystallization to be Triclinic, which Websky corroborates by his measurements.

The difficulties which are presented in the determination by measurements are two-fold. In the first place, the angles which the three most prominent faces make with each other are very closely right angles, thus suggesting one of the three orthometric systems; and, secondly, these angles are rendered uncertain by the presence of striations, a habit almost invariable. Minute crystals (Figs. 1 and 2), varying from three to five hundredths of an inch in length, were employed in the following determination, and these were taken from the surface of a pure white specimen of Cryolite. By mounting a large number of crystals, it was observed that the angles made by the three pinacoid planes with each other were quite constant, — the greatest difference from the means amounting to about  $3'$ , — so that all suspicion which I entertained as to the probability of their being right angles, was removed. A few colorless transparent crystals, absolutely free from striæ, and showing no evidence of twinning, were found, and from these the fundamental angles used in calculating the elements of the crystalline form were taken. They are represented in Fig. 1. The plane (001) was chosen for the basal section, because it formed the termination of the crystals, and was not commonly striated.

## CRYSTALLINE FORM—TRICLINIC.

Forms {100}, {010}, {001}, {1 $\bar{1}$ 0}, {201}  
 {111}, {1 $\bar{1}$ 1}, {1 $\bar{1}$ 1}, {111}, and {311}

## FUNDAMENTAL ANGLES.

Between normals	(100)	and	(010)	—	91°	53'	30"
	(100)	„	(001)	—	89°	47'	50"
	(010)	„	(001)	—	89°	55'	30"
	(111)	„	(001)	—	54°	10'	
	(111)	„	(010)	—	55°	42'	30"

From these were calculated:—

Brachydiagonal  $a = 1$

Macrodiagonal  $b = 1.00362$

Vertical axis  $c = 1.00185$

Angles between axes  $X Y = 88° \quad 6' \quad 30''$

$X Z = 90° \quad 12' \quad 20''$

$Y Z = 90° \quad 4' \quad 33''$

Angles between normals.	Measured.	Calculated.	Websky.
(100) and (010)	— 91° 53' 30"	. . .	91° 57'
(100) " (001)	— 89° 47' 50"	. . .	89° 36'
(010) " (001)	— 89° 55' 30"	. . .	89° 58'
(111) " (001)	— 54° 10'	. . .	
(111) " (100)	— 55° 42' 30"	. . .	
(111) " (100)	— 55° 31'	55° 27' 57"	
(1 $\bar{1}$ 1) " (001)	— 55° 40' 13"	55° 36' 13"	
(1 $\bar{1}$ 1) " (100)	. . .	53° 16'	
(001) " (1 $\bar{1}$ 0)	— 89° 52'	89° 54' 20"	
(001) " (201)	— 63° 20' 59"	63° 19' 43"	
(201) " (100)	— 26° 24' 22"	26° 28' 7"	
(111) " (311)	— 29° 57' 50"	30° 7' 30"	
(311) " (100)	— 25° 24' 30"	25° 25' 31"	
(311) " (001)	— 72° 2' 30"	72° 5' 17"	
(100) " (1 $\bar{1}$ 0)	— 44° 1' 54"	43° 57' 18"	
(1 $\bar{1}$ 0) " (0 $\bar{1}$ 0)	— 44° 4' 38"	44° 9' 12"	
(001) " (1 $\bar{1}$ 1)	— 54° 18' 35"	54° 25' 43"	

Fig. 2 shows the distribution of planes upon a second set of crystals which were taken from the same specimen of Cryolite as those repre-

sented in Fig. 1. These I consider twins, for the following reasons:— Faces of the form  $\{001\}$  were striated parallel to the combination edge  $(111) \wedge (\bar{1}\bar{1}1)$ , although but very faintly. The striation was shown best by a spectrum which appeared when a crystal was adjusted for measuring the angle between the poles  $(001)$  and  $(111)$ , and this spectrum did not appear when the crystal occupied any other position. Furthermore, if we assume for the moment that the drawing, Fig. 2, is that of a simple crystal, the angle between the poles of  $(0\bar{1}0)$  and  $(001)$  will be  $90^\circ 4' 30''$ , or the supplement of that between  $(010)$  and  $(001)$ . In fact, however, the following are the actual measurements:—

$$\begin{array}{lcl} (100) \wedge (001) = 89^\circ 44' 30'' & \} & (100) \wedge (\bar{1}00) \\ (001) \wedge (\bar{1}00) = 90^\circ 3' & \} & 179^\circ 47' 30'' \\ (010) \wedge (001) = 89^\circ 51' 30'' & \} & (010) \wedge (0\bar{1}0) \\ (001) \wedge (0\bar{1}0) = 89^\circ 46' & \} & 179^\circ 37' 30'' \end{array}$$

If, then, we consider the twinning-plane parallel to the form  $\{110\}$  (not present on the crystals), by turning one half of the crystal through  $180^\circ$ , the angle  $(0\bar{1}0) \wedge (001) = 89^\circ 46'$  will correspond to  $(100) \wedge (001) = 89^\circ 44' 30''$ . Again, the angle  $(\bar{1}\bar{3}1) \wedge (\bar{1}\bar{1}1)$  is the same as that found for  $(311) \wedge (111)$ . In other words, the plane, which appears in Fig. 2 as  $(\bar{1}\bar{3}1)$ , is the other member of the form  $\{311\}$ , and lies opposite  $(311)$ .

The form  $(2\bar{2})$  of Dana corresponds to what I call  $\{311\}$ . In no instance were four similar planes of the form  $\{311\}$  to be seen on any termination.

In our projection of the poles of the faces, Fig. 3, the great circles  $[001, 100]$  and  $[010, 100]$  sensibly coincide with the diameters of the circle of the primitive.

The drawing, Fig. 2, was made on the assumption that such crystals were simple, and not twinned, as I have above described them.

#### WHITE TOURMALINE.

Specimens of White Tourmaline, from Dekalb, St. Lawrence Co., N. Y., have been recently put on exhibition in the Mineral Cabinet of Harvard College, and, being interested to know what planes were represented on the crystals, I undertook the study of them. The rhombohedral forms which were observed on the several crystals are shown in Fig. 4, and also the table.

	Miller.	Bravais-Miller.	Naumann.
Rhombohedrons	{211}	{10 $\bar{1}$ 4}	$+\frac{1}{4}R$
	{110}	{1 $\bar{1}$ 02}	$-\frac{1}{2}R$
	{100}	{10 $\bar{1}$ 1}	$+R$
	{11 $\bar{1}$ }	{2201}	$-2R$
Scalenedrons	{310}	{3 $\bar{1}$ 24}	$\frac{1}{4}R\ 3$
	{21 $\bar{1}$ }	{32 $\bar{1}$ 2}	$\frac{1}{2}R\ 3$
	{312}	{5322}	$\frac{1}{2}R\ 5$
Prisms	{2 $\bar{1}$ 1}	{10 $\bar{1}$ 0}	$\infty\ P$
	{10 $\bar{1}$ }	{2 $\bar{1}$ 10}	$\infty\ P\ 2$
	{213}	{54 $\bar{1}$ 0}	$\frac{\infty\ P\ 5}{2}$

In all crystals the three prisms above tabulated were present, but the alternate planes of {2 $\bar{1}$ 1} — or the planes of  $k\{2\bar{1}1\}$ , if we regard the form {2 $\bar{1}$ 1} composed of  $k\{2\bar{1}1\}$  and  $k\{11\bar{2}\}$  — were far less prominent than the remaining planes of the same form. Indeed, they were frequently diminished to such an extent that they appeared almost as lines, and then no image could be obtained from them.

Among the crystals which were studied, one only was found doubly terminated. The forms were as follows:—

Analogue Pole.	Antilogue Pole.
{110}	$k\{211\}$
{100}	{110}
$k\{11\bar{1}\}$	{100}
$k\{312\}$	

One crystal was very highly modified, and exhibited all the forms, with the exception of {21 $\bar{1}$ }.

The following measurements were obtained: Fundamental angle (between normals) (100) and (010) =  $77^\circ 17'$ ;  $77^\circ$  Dana.

The angle between the axes of Miller's system,  $79^\circ 36'$ , or the vertical axes of Naumann's system  $c = 0.90146$ ;  $0.89526$  Dana.

Angles between normals.	Observed.	Calculated from (100) $\wedge$ (010).	Dana.
(11 $\bar{2}$ ) and (110)	$62^\circ 23'$	$62^\circ 30\frac{2}{3}'$	$62^\circ 40'$
(11 $\bar{2}$ ) " (11 $\bar{1}$ )	$25^\circ 33'$	$25^\circ 39\frac{3}{4}'$	$25^\circ 49'$
(110) " (101)	$47^\circ 6'$	$47^\circ 7\frac{1}{2}'$	$46^\circ 52'$
(110) " (211)	$23^\circ 33'$	$23^\circ 33\frac{3}{4}'$	$23^\circ 26'$
(110) " (310)	$21^\circ 53'$	$21^\circ 47\frac{1}{2}'$	

Angles between normals.	Observed.	Calculated from (100) $\wedge$ (010).	Dana.
(110) " (21 $\bar{1}$ )	29° 8	29° 10	29°
(110) " (31 $\bar{2}$ )	41° 51	41° 48	41° 48
(110) " (10 $\bar{1}$ )	66° 16	66° 26 $\frac{1}{3}$	66° 34
(31 $\bar{2}$ ) " (11 $\bar{1}$ )	21° 12	21° 19 $\frac{1}{3}$	
(11 $\bar{2}$ ) " (21 $\bar{3}$ )	11°	10° 53 $\frac{2}{5}$	10° 54
(21 $\bar{3}$ ) " (10 $\bar{1}$ )	19° 7	19° 6 $\frac{2}{5}$	19° 6
(10 $\bar{1}$ ) " (1 $\bar{1}$ 0)	59° 57	60°	60°

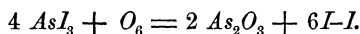
Crystals are stout, and have a section in general triangular. A perfect and easy cleavage parallel to {10 $\bar{1}$ } can be obtained, the lustre of which is pearly. Prism faces, except sometimes those of {21 $\bar{3}$ }, are remarkable for the absence of striations. The color of the mineral varies slightly: some crystals are almost pure white; others very light amber, and these are transparent. The mean specific gravity of two determinations gave the figures 3.01589. Fuses easily to a white glass, and when decomposed with the mixture of bisulphate of potassium and fluor-spar, imparts to the flame a green color, far more intense than I have previously obtained with Tourmaline.

### IODIDE OF ARSENIC.

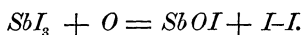
In 1880 I undertook the study of the physical and chemical properties of iodide of arsenic. Professor J. P. Cooke had previously shown (Proceedings of the American Academy, Vol. XIII.) that solutions of iodide of antimony in bisulphide of carbon were oxidized when exposed to sunlight, provided that air had free access. From the analogy of the properties of arsenic and antimony, it was thought that the iodides of these elements would exhibit the same behavior. My observations in this investigation I have recorded below.

Iodide of arsenic is very easily prepared by shaking up in a flask a solution of iodine in bisulphide of carbon with pulverized arsenic. By repeated crystallizations from bisulphide of carbon, the resulting iodide is purified, and is finally deposited in yellow-red hexagonal tables. Since iodide of arsenic is more soluble in bisulphide of carbon than iodide of antimony, the conditions are more favorable for obtaining large and stout crystals than in the case of  $SbI_3$ . Solutions of  $AsI_3$  in bisulphide of carbon are light red, but rapidly change in sunlight, the oxidation closely resembling that of  $SbI_3$ . Free iodine is given off, but remains dissolved in the bisulphide of carbon, coloring the solution dark purple. Where the solution of iodide of arsenic has moistened the

walls of the flask or containing vessel during the process of oxidation, minute octahedrons of arsenious oxide are deposited. A white deposit is also formed at the bottom of the flask, but in quantities too small to test. This deposit is probably  $As_2O_3$ . Now, this decomposition, unlike that of  $SbI_3$ , is complete, and the chemical reaction may be expressed by the formula, —



The reaction for the oxidation of  $SbI_3$  in a similar way, as given in the paper above alluded to, is as follows: —



The oxidation of iodide of arsenic, therefore, goes a step farther, so that instead of oxi-iodide, there is formed an oxide of arsenic.

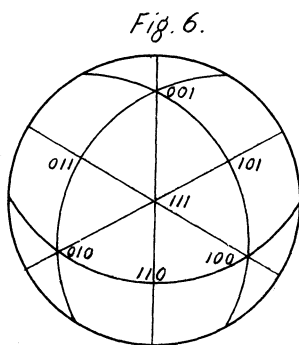
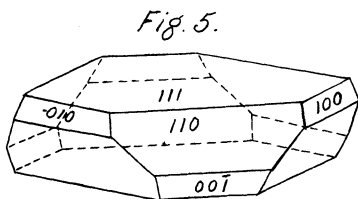
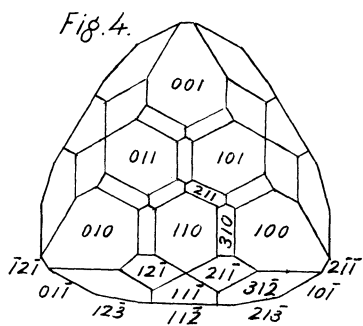
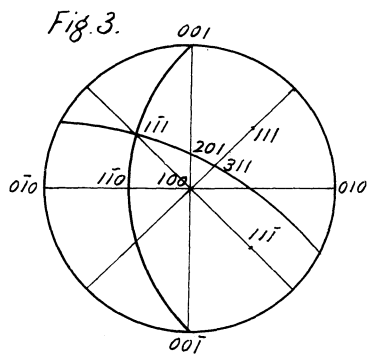
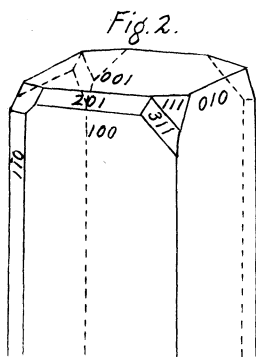
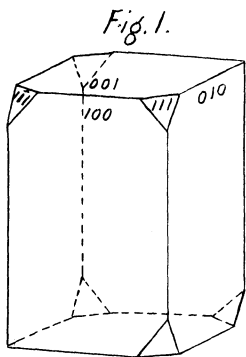
After the solution has been filtered from the oxide, distilled several times with fresh bisulphide of carbon, and then allowed to crystallize, no new modification of  $AsI_3$  could be detected. In like manner, there is no variety corresponding to the yellow trimetric iodide of antimony. Iodide of arsenic sublimes easily in yellow-red leaves, with an hexagonal outline, which, when examined with the polariscope, exhibit the phenomena of optically uniaxial crystals, with a negative double refraction.

The melting point of iodide of arsenic is  $138\frac{1}{2}^{\circ}$ – $139^{\circ}$ , about  $28^{\circ}$  lower than that of  $SbI_3$  (hexagonal variety), which is given  $167^{\circ}$ .

Iodide of arsenic crystallizes in the hexagonal system, and is isomorphous with  $SbI_3$  (red variety). The crystals of both substances consist of a rhombohedron modified by the first acute rhombohedron and basal plane, and parallel to this latter form perfect cleavages are very easily obtained. The crystals which I measured were prepared by the method above described, and also by crystallizing a German preparation of the same iodide from bisulphide of carbon.

Forms	$\{100\}$	$\{1011\}$	$+ R$	}	Figs. 5 and 6.
	$\{110\}$	$\{1102\}$	$-\frac{1}{2} R$		
	$\{111\}$	$\{0001\}$	$o P$		

Faces of the form  $\{110\}$  were more perfectly developed than those of  $\{100\}$ ; in consequence of this the fundamental angle  $(110) \wedge (111)$  was taken for calculation. Fundamental angle  $110 \wedge 111 = 59^{\circ} 48' 27''$ . The axes of Miller's system make with each other the angle  $51^{\circ} 38'$  ( $54^{\circ} 40'$  for iodide of antimony); the vertical axis of Naumann's system  $c = 2.9796$  ( $c = 2.769$  for  $SbI_3$ ). In the following summary of





angles I have compared those given by P. Friedlander, *Zeitsch. f. Kryst. u. Min.*, iii. 214.

Angles between normals.	Measured.	Calculated.	Friedlander.
(110) and (111)	59° 48' 27"	. . .	59° 59'
(110) " (001)	46° 21' 50"	46° 24' 46"	
(001) " (111)	73° 50' 47"	73° 46' 47"	
(100) " (010)	. . .	112° 31'	
(100) " (110)	. . .	56° 15' 30"	
(110) " (011)	. . .	96° 55' 30"	96° 54' (97° 10' calculated).

Faces of the form {100} were very frequently striated, but still not enough to materially vitiate the angles obtained from them, as shown by the table.

Twins are very common, the twinning plane parallel to the basal section.

I have in progress a comparison of the three iodides of antimony, arsenic, and bismuth, and the results will be published as soon as crystals of iodide of bismuth can be prepared large enough to measure.